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1 **Assessment of the point source impact pollution from the wastewater discharge into the**
2 **Danube River in the city of Novi Sad, Serbia**

3
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5
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10
11 **Abstract**

12 On-line monitoring was performed using spectrolyser equipment, coupled with laboratory
13 analysis for samples collected from wastewater discharge in the city of Novi Sad, Serbia,
14 during first 24h of three and 48h of six monitoring campaigns from December of 2012 to
15 April of 2013. Significant correlation with $R^2 > 0.9$ was observed between laboratory analysis
16 and spectrolyser measurements. COD/BOD₅ ratio in combined industrial and municipal
17 wastewater ranged from 1.2 to 2.0 indicating the presence of biodegradable organic matter
18 which could be easily removed using aeration treatment process. Micro/trace element and/or
19 heavy metals in wastewater samples were within the limits as per the standard prescribed for
20 wastewater, and should not pose any serious hazard risk. However BOD, COD, ammonia and
21 total phosphorus concentrations were measured above the limit value according to Serbian
22 and EU legislation and should be reduced before discharging wastewater directly into the
23 Danube River.

24
25 **Keywords:** spectrolyser, chemical and biological oxygen demand, wastewater

26
27 **1. Introduction**

28 The quality of the surface water is a significant problem in many regions of the world. It can
29 frequently limit the use of this essential resource and, in more extreme circumstances, harm
30 human health, biota and environment. Some studies of water quality in various effluents
31 (Meybeck 2002; Hanrahan et al. 2003; Astel et al. 2007; Simeonov et al. 2003; Dawe 2006)
32 revealed that anthropogenic activities have an important negative environmental impact on
33 water quality in the downstream sections of the major rivers. This is a result of cumulative
34 effects from upstream development but also from inadequate wastewater treatment (Popa et
35 al. 2012). Water quality decay, characterized by important modifications of chemical oxygen
36 demand (COD), total suspended solids (TSS), total nitrogen (TN), total phosphorous (TP),
37 iron (Fe), nickel (Ni), copper (Cu), zinc (Zn), lead (Pb), and so forth are the result of
38 wastewater discharge into the rivers (Popa et al. 2012). Wastewater discharges from sewage
39 and industries are major components of water pollution, contributing to oxygen demand and
40 nutrient loading of the water bodies, leading to a destabilized aquatic ecosystem (Morrison et
41 al. 2001).

42 Recent environmental legislation implies the need for optimization of wastewater treatment
43 and, therefore, a need for tools which provide quantitative and qualitative information on
44 municipal wastewater characterization and evolution (Vaillant 2002). One of main tasks
45 conducted by European Water Framework Directive (WFD) (Petrescu et al. 2012) is
46 continuous monitoring of the water regime, together with the determination of the water
47 quantity and quality, which provides a relevant source of information in presenting the state of

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48 water resources in real time. Also, the analysis of the surface water quality is fundamental for
49 a sustainable management of water resources. Even though, the directive does not introduce
50 innovative devices for quality control as well as on-line measurements, automated
51 technologies contribute to improve monitoring of water in terms of a new approach to water
52 monitoring. Moreover, on-line monitoring allows the implementation of policies intended to
53 reduce pollution load, improve controls on industrial discharges into the network (Thomas
54 and Pouet 2006) and, as a result of reducing the overall pollution burden, protecting the
55 WWTP and network equipment. Accidental discharges can have a damaging effect on the
56 biological processes involved in wastewater treatment and if not adequately treated, can pass
57 through the treatment system and into the receiving waters where it can have a harmful effect
58 on the environment and eventually reach the sources of potable water supplies causing taste
59 and odor problems in the supply of drinking water (Bourgeois et al. 2003).

60 Within the framework of research, for the first time in the city of Novi Sad, continuous optical
61 on-line monitoring system was introduced using Spectrolyser device (S::CAN, Austria,
62 Vienna) for *in situ* measurements, which allows continuous measurement of basic
63 physicochemical characteristics of wastewater (BOD₅, COD, NO₃-N, TSS, SAC254 and
64 SAC436) at each measuring point. Data obtained by these measurements were complemented
65 with an accredited laboratory analysis, in order to complete observation of the Danube raw
66 water real status on the selected site. During the measurement campaign on-line sampling in
67 winter 2012/13 and spring 2013 (December - April) samples were collected three times a day,
68 for a period of 24h of three and 48h of six monitoring campaigns. Analysis of the basic
69 physico - chemical parameters and the concentrations of metals was performed in accredited
70 Laboratory for monitoring of landfills, waste water and air.

71

72 **2. Material and methods**

73 On-site monitoring was performed using s::can spectro::lyser (S::can Messtechnik, Vienna,
74 Austria) on sampling site GC 2 (19° 51' 25,139" E, 45° 15' 44,581" N), which is one of four
75 sewage discharges in Novi Sad where the greatest amount of industrial wastewater is
76 discharged directly into Danube river, indicating the greatest impact on the variation of the
77 Danube river pollution. Basin pump station GC2 covers the northern half of the city (size
78 about 900 ha). In this area there are about 100,000 inhabitants.

79 Continuous measurement of basic physicochemical characteristics of wastewater (BOD₅,
80 COD, NO₃-N, TSS, SAC254 and SAC436) was performed every 15 min. Nine sampling
81 campaigns were conducted during five months in winter 2012/13 and spring 2013. During
82 first 24h and 48h of continuous monitoring samples for laboratory analysis were collected
83 three times per day at 6:00h, 14:00h and 22:00h. Data for average daily temperature, humidity
84 and precipitation during nine sampling campaigns in 2012/13 were obtained from Republic
85 Hydrometeorological Service of Serbia.

86 Wastewater samples for laboratory analysis were collected from GC 2 sampling site in the
87 city of Novi Sad in sealed 1L glass bottles, stored in hand refrigerator at 4 °C, and transported
88 to the laboratory. Samples were analyzed for oxygen (BOD₅, COD, dissolved oxygen,
89 permanganate index), nutrients (ammonium, nitrite, nitrate, total phosphorus), conductivity,
90 sulfate concentrations and the concentrations of metals: Pb, Fe, Cd, Cr, Ni, Zn, according to
91 the Standard Methods for the Examination of Water and Wastewater (Eaton and Clesceri
92 2005). Conductivity and dissolved oxygen were determined *in situ* using portable Multi 340i
93 WISSENSCHAFTLICH – TECHNISCHE WERKSTATTEN GMBH device. Biological
94 oxygen demand (BOD₅) was determined using the BODTrak™ method. The HachBODTrak
95 apparatus is based on the manometric principle of operation. The chemical oxygen demand,
96 ammonium, nitrite, nitrate, total phosphorus and sulfate concentrations were measured with

97 UVVIS spectrophotometer (DR 5000, HACH, Germany). In order to verify the precision and
 98 accuracy of the methods, the certified reference materials Demand WP, Simple nutrients WP,
 99 Complex nutrients WP (RTC, UK) have been used.

100 For the determination of metals, wastewater samples were subjected to digestion, spiked with
 101 5ml of HNO₃ using the microwave assisted digestion system MWS-3+ (Berghof, Germany)
 102 and analyzed using Thermo atomic absorption spectrometer with hollow cathode lamp and a
 103 deuterium background corrector, at respective resonance line using an air-acetylene flame
 104 with addition of nitrogen suboxide flame for Cr. The recoveries were carried out by the
 105 addition of the standards of each element at different levels. Blanks were included in each
 106 batch of analysis. Accuracy was evaluated with the certified reference materials LGC6175
 107 (LGC, UK) and SPS-WW2 Batch 110 (SPS, Norway). Recoveries ranged from 89 % to 97 %.
 108 Calibration curves for the determination of metals were processed with different dilutions of
 109 the standard stock solutions conc. 1000 mg/l (J.T: Baker, The Netherlands); the linear
 110 regression lines were obtained with R₂≥0.95.

111 The Data were processed using software ana::pro for s::can spectro::lyser. Correlations
 112 between the data obtained in on-line monitoring and laboratory analysis for BOD₅ and COD
 113 concentrations were analyzed using Pearson's correlation coefficients by IBM SPSS software
 114 (a significance threshold of p=0.01 was retained).The influence of meteorological data on
 115 pollutant concentrations in wastewater was also evaluated using IBM SPSS software.

117 3. Results and discussion

118 3.1. Laboratory results

119 The concentration ranges of basic physicochemical parameters and metals were presented in
 120 table 1. The highest concentrations were obtained for ammonia, biological and chemical
 121 oxygen demand. Electrical conductivity ranged from 568 to 4450 µS/cm, indicating the high
 122 salinity of wastewater. The limit value for total phosphorus (2 mg/l) and total nitrogen (15
 123 mg/l) according Serbian and EU legislation has been exceeded in the most of analyzed
 124 samples since ammonia concentrations were in range 13.7 – 60.4 mg/l.

125
 126 Table 1. Concentration ranges of metals and basic physicochemical parameters in wastewater
 127 samples (n=43)

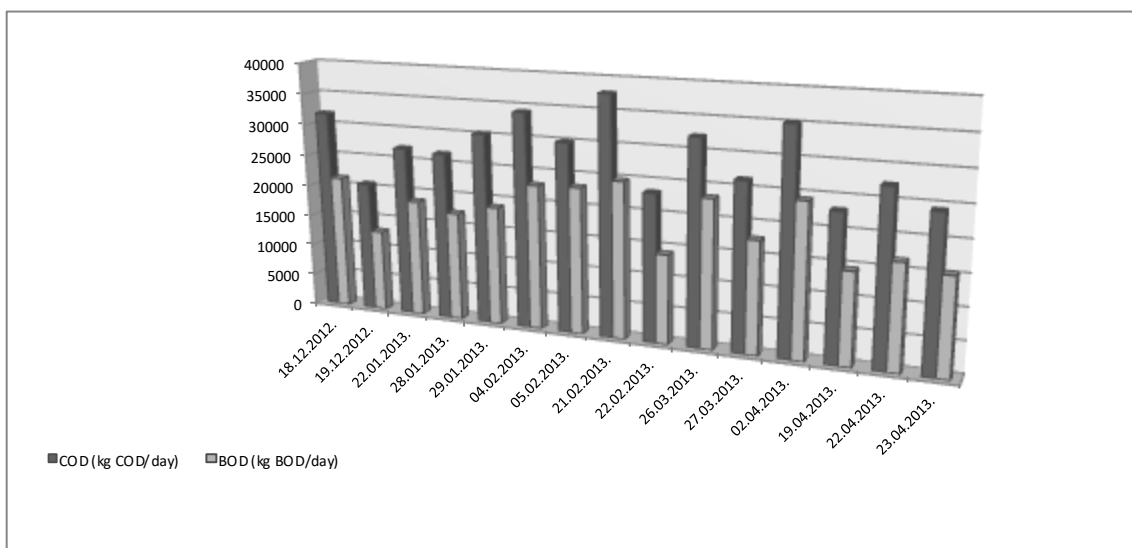
Parameter	Min-Max	Mean±SD
Conductivity (µS/cm)	568-4450	1362±746
D.O. (mg/l)	0.07-3.57	0.90±0.72
BOD ₅ (mg/l)	100-614	369±134
COD(mg/l)	196-839	556±192
NO ₃ ⁻ (mg/l)	0.1-0.5	0.3±0.1
NO ₂ ⁻ (mg/l)	0.01-0.17	0.05±0.04
NH ₃ (mg/l)	13.7-60.4	37.8±11.1
Total P (mg/l)	1.6-8.7	4.6±1.2
SO ₂ ⁻⁴ (mg/l)	35-128	63±22
Permanganate index (mg/l)	10.1-81.1	48.1±16.8
Cr (mg/l)	<0.1	
Pb (mg/l)	0.01-0.15	0.06±0.03
Fe (mg/l)	0.3-9.9	1.5±1.7
Cd	<0.005	
Ni	0.04-0.12	0.09±0.02
Zn	0.03-0.51	0.17±0.11

128 The analytical measurement data for Cr and Cd were under limits of detection (LOD) in all
129 mixed wastewater samples. The analysis for micro/trace element and/or heavy metals in
130 mixed wastewater samples shows that Zn, Fe, Pb, Cd, Cr and Ni are well within the limits as
131 per the standard prescribed for communal wastewater and should not pose any serious hazard
132 risk.

133 Average daily values of COD and BOD₅ were 556±192 and 369±134 mg/l, respectively.
134 Directive Council Directive (91/271/EEC) and the Regulation on limit values of pollutants in
135 water and deadlines for their achievement (Official Gazette of the Republic of Serbia, No.
136 67/2011 and 48/2012) prescribes threshold limit values for the parameters COD and BOD₅ in
137 the wastewater discharged into the surface waters as 125 and 25 mg/l, respectively. COD and
138 BOD₅ were measured in all mixed wastewater samples above the prescribed limits.

139 The reduction of BOD₅ and COD in the separate treatment units of a plant can be used to
140 measure the efficiency of each unit in the wastewater treatment process. It is very important to
141 calculate the ratio of COD/BOD₅ because it indicates the biodegradability of wastewater. The
142 higher the ratio is the less the biodegradability of the wastewater. Ratio values depend on the
143 nature of the wastewater and the COD/BOD₅ ratio value for municipal raw wastewater is in
144 the range from 1.25 to 2.5, whereas for industrial wastewater the ratio goes up to 10 or more.
145 COD/BOD₅ ratio in combined industrial and municipal wastewater samples analyzed in this
146 study ranged from 1.2 to 2.0 indicating the higher quantity of municipal wastewater compared
147 to industrial wastewater and therefore high ability of wastewater to biologically degrade.

148 Total organic load of the wastewater at discharge GC 2 in kg COD/day and kg BOD/day was
149 calculated on the basis of the average daily flows data (540 – 800 l/s) and results were shown
150 in figure 1. COD loads ranged from 20731 to 37809 kg/day, while BOD loads of wastewater
151 were in range 13033 – 24791 kg/day.



152
153 Figure 1. Total daily organic load of the wastewater at discharge GC 2
154

155 3.2 Continuous monitoring results

156 Data were obtained for COD, BOD, SAC254 and SAC436 every 15 minutes in all sampling
157 campaigns. Studies have shown that during stable temperature and precipitation conditions
158 SAC parameters are compatible with COD and BOD parameters. It can be concluded that
159 COD and SAC254, and BOD and SAC 436 are compatible with the same trendline and
160 fluctuations, respectively. For the period from January 28th until February 2nd 2013, according
161 to data obtained from Republic Hydrometeorological Service of Serbia, temperature and
162 precipitation were higher than normal for that part of the year, which can be noticed by

163 diverging of trendlines of parameters for mentioned sampling period. High temperature
164 (extreme) and normal precipitation levels for sampling campaign in April have shown an
165 interesting and unexpected behavior of measured parameters. During unstable weather
166 conditions values of parameters COD/BOD/SAC were drastically varying. Online monitoring
167 curves for BOD₅ and COD showed periodic trends with peak minimum between 4:00 h and
168 7:00 h and two peaks maximum between 13:00 h and 17:00 h as well as 20:00 h and 00:00 h.
169 NO₃⁻ concentrations were under limit of detection (LOD) in all sampling campaigns.
170 For overall data from 5 months period of monitoring it can be noticed that trendlines (R²) and
171 shape of the parameters curve for COD/BOD and SAC correspond. Two correlations can be
172 noticed CODEq/SAC436 and BODEq/SAC254. SAC parameters represent communal organic
173 load of water, while CODEq and BODEq detect industrial and agricultural organic load in the
174 effluent, respectively. As the sewerage system of Novi Sad is mixed sewerage system
175 collecting communal, industrial and small amount of agricultural wastewater; the main
176 conclusion from data evaluation that can be derived for monitoring period is that SAC
177 parameters show 'clean' flow of communal wastewater organic load, while CODEq and
178 BODEq have periodical picks (extremes) that can be only explained as human behavior
179 routines, as well as high load of industrial and agricultural effluents to sewerage system,
180 respectively.

181

182 *3.3 Correlation between online monitoring and laboratory analysis*

183 Statistical analysis of obtained results indicated good correlation between on-line continuous
184 measurements and laboratory analysis. Within nine sampling campaigns during winter period
185 2012/13 and spring 2013, linear curves were obtained for COD and BOD₅ with R² of 0.931
186 and 0.903, respectively. Pearson correlation coefficients (r) showed positive correlations with
187 values 0.910 (p=0.01) and 0.657 (p=0.01) for COD and BOD₅, respectively. Better correlation
188 was obtained for COD, because of difference in measuring BOD. Using on line measuring
189 equipment obtained values for BOD are for every moment of time and in laboratory
190 conditions the BOD₅ levels were determined. The total suspended solids (TSS) concentrations
191 were not measured in laboratory conditions, so measuring data are obtained using S::can UV
192 probe. These results were not included into the evaluation process, during this particular
193 study.

194

195 *3.4. The impact of seasonal condition variations and meteorological parameters on the* 196 *quality of wastewater*

197 The analysis of the seasonal variations and the meteorological parameters influence on the
198 quality of the wastewater showed a significant effect of humidity on the dissolved oxygen
199 concentrations (Table 2). The results obtained for dissolved oxygen showed daily variations
200 that may result from activities of aquatic organisms; the minimum is registered in the early
201 morning hours and maximum in the early afternoon when the water temperature is the
202 highest. The concentration levels of sulfate showed the dependence of the air and water
203 temperature while a concentration of iron depends just on the levels of a water temperature.
204 Precipitation has a significant effect on the majority of measured values of electrical
205 conductivity, dissolved oxygen, ammonium, COD and total phosphorus. It could be
206 concluded that seasonal variations such as changes in precipitation and changes in air and
207 water temperatures have a significant impact on the quality of wastewater. Correlation matrix
208 with the values of the Pearson coefficients with highlighting the significant correlations with
209 the probability of 95% and 99% are shown in Table 2.

210

211 Table 2. Correlation matrix of the influence of meteorological parameters on the quality of
 212 wastewater

	Conduct.	D.O.	NH ₃	Total P	Perm. index	COD	BOD ₅	Sulfate	Fe
Air temp.	-0.252	-0.262	0.019	-0.035	0.397	-0.143	-0.254	0.761(**)	-0.434
Humidity	0.424	0.555(*)	-0.460	-0.372	-0.005	-0.147	-0.133	-0.392	0.396
Precipitation	0.779(**)	0.718(**)	-0.614(*)	-0.549(*)	-0.217	-0.554(*)	-0.452	-0.287	0.439
Water temp.	-0.453	-0.460	0.376	0.244	0.204	0.087	-0.035	0.648(**)	-0.611(*)

213 (**) Correlation is significant at the level $p = 0.01$

214 (*) Correlation is significant at the level $p = 0.05$

215
 216 Average monthly concentrations of selected parameters in the effluent during the sampling
 217 period from December of 2012 until April of 2013 were calculated and the next conclusions
 218 were derived. In the campaign conducted in February, lower concentrations values of various
 219 parameters were observed in comparison with previous campaigns in December and January
 220 as a result of dilution effect due to melting of snow. In March, during the period of highest
 221 rainfall and high humidity, conductivity reached a maximum value of two times greater than
 222 in other months of the campaign, while the minimum value was measured for phosphorus
 223 content. In April of 2013, during the dry period of the year with the lowest humidity and
 224 highest temperatures in comparison with the previous series of measurements, the
 225 concentration of sulfate reached a maximum value.

226 227 4. Conclusions

228 Data obtained within the monitoring program provide insight into detailed influence of mixed
 229 urban (industrial and communal) and agricultural wastewater discharges onto the Danube
 230 River surface water quality, which contributes to better identification of the pollutant loads,
 231 creating the basis for risk assessment and ultimately establishing standards of chemical
 232 quality, as well as a good basis for future wastewater treatment plant design and
 233 improvement. The great importance of this study is reflected in the fact that continuous on-
 234 line monitoring of wastewater using an optical system, Spectrolyser device is introduced for
 235 the first time in the city of Novi Sad, providing continuous measurements of basic physical
 236 and chemical characteristics of wastewater (BOD₅, COD, TSS, SAC254, SAC436) at each
 237 time point. The data obtained in this way are supplemented by laboratory analysis using
 238 standard testing methods in order to get more comprehensive review of the real status of
 239 wastewater discharged directly into Danube River on the selected site. Results of laboratory
 240 analysis parameters of biological and chemical oxygen demand indicated good correlations
 241 with the results obtained with on-line measurement, with $R^2 > 0.9$, thus confirming the
 242 efficiency of continuous measurement with spectrolyser equipment. It could be concluded that
 243 this equipment as time and cost effective tool can be used for continuous on-line monitoring
 244 of communal wastewater.

245 The study clearly indicates that some of the physicochemical and heavy metal parameters
 246 undertaken to assess the water quality of wastewater discharge were found below the
 247 prescribed limit (Zn, Cd, Cr and Ni), which indicates a low risk level for negative

248 environmental effects induced from occurrence of toxic metals, while some parameters were
249 measured above the appointed regulation limits (BOD₅, COD, ammonia, total phosphorus).
250 COD/BOD₅ values indicating the presence of higher biodegradable organic matter which
251 could be effectively removed with appropriate design of aeration treatment process. High
252 biodegradability of wastewater according to COD/BOD₅ ratio improves the efficiency of
253 subsequent biological treatment.

254 It is well known that wastewater is a huge problem, even in developed countries, for
255 environmental protection from chemical and other sanitary risks to human health. That is the
256 reason why research on wastewater quality has to be encouraged with development of on-site
257 measurement methods. The design and implementation of future monitoring programs of
258 wastewater in the city of Novi Sad using on-line device as early warning system, would give
259 information that will enable timely, effective and appropriate actions to prevent further
260 degradation of the Danube River surface water quality and possible negative effects on
261 aquatic life. The obtained monitoring data will be used for implementation of modern
262 approach to wastewater treatment processes and plant design as a principle of good practice
263 and to propose the most appropriate method for collection, transport, treatment and
264 discharging of effluent into the recipient, the Danube River.

265

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271

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